# Standard Operating Procedure

for

Routine Operation of the Sequential Gas Sampler (SGS)

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Simultaneous Collection of Gases and PM<sub>2.5</sub> or PM<sub>10</sub>

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### 1. GENERAL DISCUSSION

### 1.1 Purpose of Procedure

This procedure describes the sampler configuration and instructions for operating the DRI MEDVOL sequential gas sampler during the Treasure Valley Secondary Aerosol Study. Suspended particulate matter in the 0 to 2.5 (PM<sub>2.5</sub>) or 0 to 10 (PM<sub>10</sub>) micrometer size ranges and gaseous species are collected on filter substrates which are subsequently analyzed for different chemical species. During operation, one set of two filter packs is used to sample air. Up to six sets of two filter packs can be collected sequentially between sample changes.

Filter media are chosen so that concentrations of mass, trace elements, ions, organic and elemental carbon, gaseous nitric acid, ammonium/ammonia, and sulfur dioxide are collected and quantified by laboratory analyses.

## 1.2 Measurement Principle

The sampler can be fitted with either a PM<sub>2.5</sub> or a PM<sub>10</sub> inlet. For the PM<sub>2.5</sub> inlet equipped sampler, particles larger than 2.5 micrometers in aerodynamic diameter are removed from the air stream with a Bendix 240 cyclone operating at a flow rate of 113 liters per minute (lpm). For the PM<sub>10</sub> inlet equipped sampler, particles greater than 10 micrometers in aerodynamic diameter are removed from the air stream with a Sierra Andersen 254 sizeselective PM<sub>10</sub> inlet operating at a flow rate of 113 lpm. The PM<sub>2.5</sub> or PM<sub>10</sub> sized aerosol is routed into a conical plenum. The conical shape of the plenum diffuses the airflow and minimizes particle deposition. Particles and gases are collected on open faced filter packs inserted in the base of the plenum. For some applications, an annular denuder is placed upstream of one or both of the open-faced filter packs; the annular denuder extends out of the bottom of the conical plenum. There are two types of annular denuders that may be used as part of the sampling configuration. An anodized aluminum oxide denuder is used to remove nitric acid gas from the sample air stream, whereas a citric acid-coated glass denuder removes ammonia gas. Filter packs are connected to vacuum pumps through switchable solenoid valves. Flow rates are controlled by differential pressure regulators that maintain constant pressure across ball valves. A programmable timer activates two vacuum pumps and switches solenoid valves for sample collection. Up to 56.5 lpm of air flow are drawn through each of two filter packs simultaneously. If additional flow is required to maintain the total flow rate of 113 lpm through the inlet, air is drawn through a makeup air tap in the base of plenum.

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The base has thirteen filter holder ports. The sampler is configured to allow a maximum of six sets of two filter packs to collect sequentially. The thirteenth and final port is plugged with a blank filter holder and is used only if additional "make up air" is required. Each filter pack can contain up to 3 separate 47 mm diameter filter substrates which collect particles and gases for later analyses. The filter holders are made by Savillex of PFA Teflon to minimize their reaction with the sample and contain redesigned filter backing trays that reduced flow restriction and provide uniform deposition. Filters that are used include PTFE Teflon membranes (e. g., Gelman (Ann Arbor, MI) polyolefin ringed, 2.0 µm pore size, (#R2PJ047)) for gravimetric and x-ray fluorescence analysis); pre-fired quartz fiber filters (e. g., Pallflex (#2500QAOT-UP)) for soluble ion and carbon analyses; cellulose filters (e. g., Whatman 31ET) impregnated with K<sub>2</sub>CO<sub>3</sub> to collect SO<sub>2</sub>; with NaCl to collect volatile nitrates, or with citric acid to collect volatile ammonia.

The sampler has a programmable timer that controls the sampling schedule Elapsed time indicators (ETI) measure the duration of each sample. A pressure-activated switch is included to turn the ETI's off if vacuum is lost in the flow system because of pump or other component failure.

### 1.3 Measurement Interferences

### 1.3.1 Passive Deposition

Passive deposition occurs when particles deposit on and gases are absorbed by filters prior to and after sampling. Field blanks are used to quantify this bias, which is usually less than 30 µg of particle mass per filter.

### 1.3.2 Inlet Loading and Re-entrainment

Material collected in the size-selective inlet can become re-entrained in the sample flow. The inlets are cleaned semi-annually to minimize overloading and re-entrainment.

### 1.3.3 Pump Exhaust Recirculation

Recirculation occurs when the pump exhaust, which contains carbon and copper particles from the pump vanes and motor armature, is entrained in the sampled air. Recirculation is minimized by re-conditioning the pumps annually, filtering exhausted air with a total filter, and locating pump exhaust inside an enclosure over one meter below the sampling inlet.

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Exhaust filters are replaced regularly. When the pumps are new or when the vanes are replaced, the pumps are allowed to run for 24-hours without a load to break-in the vanes and lessen the subsequent wear on the vanes.

#### 1.3.4 Particle Volatilization

Ammonium nitrate can dissociate and the particulate nitrate can escape as nitric acid gas. This effect can be quantified by placing a NaCl impregnated cellulose backup filter behind a quartz filter to absorb volatilized nitric acid. The PFA Teflon Savillex filter holder has been shown to be inert to nitric acid absorption and can accommodate multiple filters. Filters are unloaded and kept under refrigeration after sampling to minimize long-term volatilization.

### 1.3.5 Filter Gas Absorption

Quartz fiber filters have been shown to absorb significant quantities of organic vapors which are measured as organic particulate carbon. A quartz backup filter can be placed behind the Teflon filter to quantify this artifact and to allow for its subtraction, if it is deemed necessary. The Savillex filter holder can accommodate such a stack. Quartz fiber filters have been shown to absorb insignificant amounts of sulfur dioxide, nitric acid, and nitrogen oxides. Nitric acid absorbed by NaCl-impregnated filters will appear as nitrate in the analysis. To avoid this problem, an aluminum annular denuder is used to remove gaseous nitric acid from the sample air.

### 1.3.6 Filter Integrity and Contamination

Filter integrity is compromised by handling which causes pieces of the filter to be lost after the pre-exposure weighing. Filter contamination results from material other than sampled aerosol and gases being deposited on the filter (e.g. fingerprints, dirt). The effects of filter material losses are minimized by performing gravimetric analysis on Teflon membrane filters which are less friable than the quartz fiber filters. Filter material losses and contamination are minimized by the placement and removal of filters to and from filter holders in controlled laboratory conditions. Gloved hands and forceps are used in this filter processing. Spare, loaded filter holders are provided in the field to eliminate the need for field loading and unloading. Each filter holder is separately sealed prior to and after sampling. Batches of filters are inspected and submitted to chemical analysis prior to use to assure that they meet adequate standards when received from the manufacturer.

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### 1.3.7 Particle Loss During Transport

Coarse particles (greater than  $2.5~\mu m$ ) have been found to be shaken from filters during transport on heavily loaded filters. Flow rates are adjusted to minimize overloading of filters. Loss of  $PM_{2.5}$  particles is insignificant.

#### 1.3.8 Transmission Losses

The necessity of particles to pass through a size-selective inlet could result in particle losses. Distances between the inlet and the filter surfaces have been minimized to reduce these potential losses, and airflow paths have been designed to minimize the chance of particle impaction.

Nitric acid reacts with most surfaces other than Teflon and glass. All sampling surfaces of the  $PM_{2.5}$  sampler, including the inlet, cyclone, bug screen and plenum are coated with PTFA Teflon to minimize nitric acid loss. Teflon tubing connects the two halves of the inlet; filter holder parts that contact sampled air are constructed of Teflon.

### 1.4 Ranges and Typical Values

The range of concentrations measured by this method depends on local air quality conditions. For mass, the concentration range is typically 5 to 300 µg/m<sup>3</sup>.

### 1.5 Typical Lower Quantifiable Limits, Precision, and Accuracy

Lower quantifiable limits and precision are species dependent, and are determined by the sensitivity of the analysis method and the variability of field blank concentrations. The mass lower quantifiable limit is about 1  $\mu$ g/m³. Mass precision is approximately  $\pm$  5% relative, for mass concentrations which exceed ten times the lower quantifiable limit. Accuracy is generally  $\pm$  10% relative, for mass determinations.

### 1.6 Responsibilities

The IDEQ field technician is responsible for carrying out this standard operating procedure and for the completion and submission of all documents. The IDEQ field operations supervisor is responsible for scheduling the field technician's visits, reviewing documentation, identifying and reviewing deficiencies, and receiving samples from and

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transmitting samples to the laboratory. The DRI laboratory supervisor is responsible for preparing filter substrates, transmitting them to the field, and receiving them from the field.

## 1.7 Definitions

DRI MEDVOL The entire sampling unit (also called a sequential gas sampler).

PM<sub>10</sub> Inlet Sierra Andersen 254 size-selective inlet.

PM<sub>2.5</sub> Inlet Two metal cylindrical containers, both open on the bottom, held side by

side with brackets. Containers are connected with an inverted "U" shaped Teflon tube through openings in the container tops. One container has a bug screen mounted on the bottom and a Bendix 240 cyclone inside. The

other container mounts on top of the plenum.

Plenum The conical chamber into which filter holders are inserted.

DDW Distilled, deionized water.

### 1.8 Related Procedures

This standard operating procedure covers all aspects of sampler operation.

Rotameter calibration is covered in SOP 2-210.1: "CADMP Sampler: Calibration of Flow Meters" dated 2nd quarter 1990.

Siting criteria are contained in "Quality Assurance Handbook for Air Pollution Measurement Systems. Volume II. Ambient Air Specific Methods", section 2.0.2.

Filter pack preparation is covered in SOP 2-208.3: "Filter Pack Assembly, Disassembly, and Cleaning Procedure."

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# 2. SEQUENTIAL GAS SAMPLER INSTRUMENTATION FOR THE TREASURE VALLEY SECONDARY AEROSOL STUDY

### 2.1 Ambient PM<sub>2.5</sub> Sampling Overview

To specifically address the data needs of the Treasure Valley study objective, three sets of sequential gas samplers (SGS) and one sequential filter sampler (SFS) will be deployed at each of the two core sites. Five 4 to 6-hour samples will be collected on twenty-one days in which high secondary aerosol concentrations are expected. The sequential filter sampler (SFS) is used to measure ambient concentrations of PM<sub>2.5</sub> mass, elemental composition, elemental and organic carbon mass, ion concentrations, as well as PM<sub>2.5</sub> nitrate. The DRI standard operating procedure for the SFS is given in the Document entitled "DRI Standard Operating Procedure: DRI Sequential Filter Sampler".

The three sequential gas samplers that will be deployed at each of the two sites are used to measure airborne concentrations of: 1) ammonia gas/total ammonia (PM<sub>2.5</sub> ammonium + ammonia gas); 2) nitric acid gas/total nitrate species (nitric acid gas + particle nitrate sampled without size cut), 3) sulfur dioxide gas.

# 2.2 Ammonia/Ammonium particle

Figures 2.1.a and 2.2 illustrate the configuration of the ammonia/ammonium sequential gas sampling system. A PM<sub>2.5</sub> PFA Teflon-coated cyclone is used at the inlet of the conical plenum. Within the conical plenum, air is sampled through two different channels, each operating at 56.5 lpm for a total flow of 113 lpm through the cyclone inlet. In the first channel, a citric acid-impregnated cellulose filter (Whatman 31ET) is used in a single stage 47 mm Savillex filter holder. The filter holder is made of injection-molded PFA Teflon, the substance which has shown the lowest inclination to absorb nitric acid or any other gaseous species. The Savillex filter support grid has been replaced with a grid manufactured by ATEC, Inc. (Calabasas, CA), which is especially designed for dry deposition monitoring. This new support grid has a more uniform porosity that results in a uniform filter deposit. It also reduces the flow resistance across the filter holder. The cellulose filter in the first channel (GOC) captures both ammonium particles and ammonia gas. In the second channel, a citric acid-coated annular denuder is placed upstream of a citric acid-impregnated cellulose filter (Whatman 31ET) (GDC) in order to remove ammonia gas from the sample air. The ammonia denuder consists of 16 hydrogen fluoride acid etched glass tubes (6 mm ID x 8 mm OD x 25 cm) that have been coated with a solution of 10% citric acid/2% glycerol/88% methanol. Ammonium found on the filter in the second channel is due primarily to ammonium particles. This method allows for calculation of ammonia particle concentrations (PM<sub>2.5</sub>) and ammonia gas concentrations (difference between total

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ammonium found on filter in Channel 1 and ammonium due to  $PM_{2.5}$  found on filter in Channel 2).

### 2.2.1 Applicability of SOP to Ammonia/Ammonium Sampling

This SOP is applicable to ammonia/ammonium sampling. While the SOP does not explicitly state what should be done in the event that a denuder is used in conjunction with a filter pack, in most cases, it is possible to simply consider the denuder as a port in the SGS sampler. E.g., if the SOP states "Remove filter pack from Ports 1 and 7 on plenum" you may assume that the equivalent statement for a denuder/filter pack combination is "remove filter pack from Port 1 on plenum and from the denuder attached to Port 7 on plenum".

# 2.3 Nitric acid gas/ particulate nitrate

Figures 2.1.b and 2.3 illustrate the configuration of the nitric acid/total nitrate sequential gas sampler. The configuration for nitric acid sampling is somewhat different than for ammonia/ammonium and sulfur dioxide sampling due to some special concerns associated with nitric acid measurement. Nitric acid deposits on just about any surface and has a high reactivity with coarse, alkaline particles. Previous nitric acid measurements have shown that the choice of sampling inlet, sampling surface, and filter holders are critical. Preliminary studies performed at DRI have shown that when sampling using a standard sequential gas sampler configuration, as much as 60% of ambient nitric acid is deposited on the PFA coated sampling plenum and the Bendix cyclone. To minimize the adsorption of nitric acid within any sampling train, the sampling of nitric acid is configured so that the sample air stream does not come into contact with the size-selective inlet or the conical plenum. As shown in Figure 2.1.b, this is accomplished by arranging filter packs and denuders on a wheel mounted above the conical plenum.

Figure 2.3 shows a schematic of the two channels used for sampling nitric acid/particulate nitrate. Channel 1 consists of a NaCl-impregnated cellulose filter (Whatman 31ET) (GON) to collect total nitrate (total particulate nitrate plus nitric acid). The same filter is used in Channel 2 (GDN), however in this case, the Savillex filter pack is preceded by an anodized aluminum nitric acid denuder to remove nitric acid from the sample air. Thus, the nitrate found on the cellulose filter in channel 2 is due primarily to particulate nitrate; Watson et al. (1991) have shown that the anodized aluminum denuder tubes remove more than 95% of gaseous nitric acid from the sample air. Nitric acid gas concentration is determined by the difference between total nitrate (particulate nitrate plus gaseous nitric acid) and particulate nitrate. Note that in this sampling configuration, the particle sizes associated with nitrate are not limited by a size-selective inlet. This system allows direct sampling of gas/aerosol with minimal residence time to ensure a high sampling efficiency for nitric acid.

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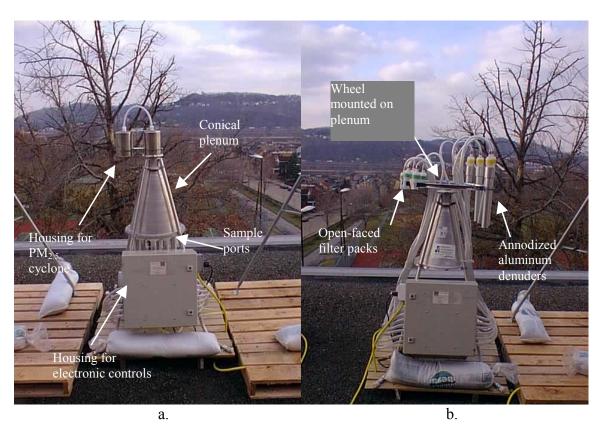


Figure 2-1 Photograph of SGS in a) standard configuration used for ammonia/ammonium and sulfur dioxide sampling, and b) modified configuration used for HNO<sub>3</sub> sampling. Note that in b) a wheel is mounted on top of the conical plenum and that a size-selective inlet is not used.

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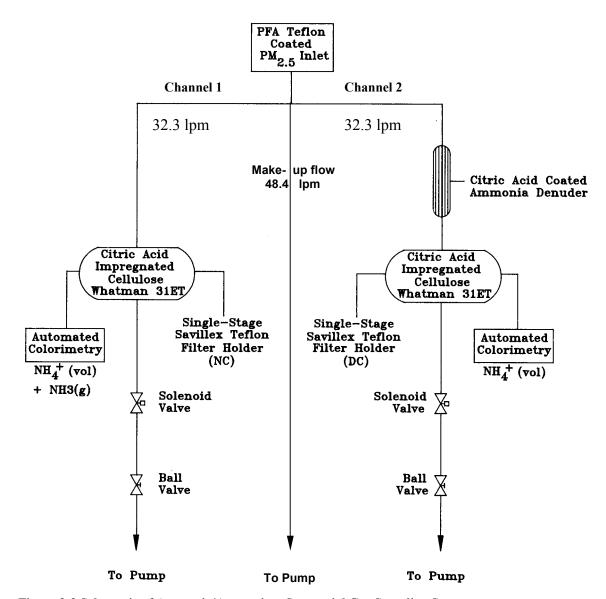


Figure 2-2 Schematic of Ammonia/Ammonium Sequential Gas Sampling System

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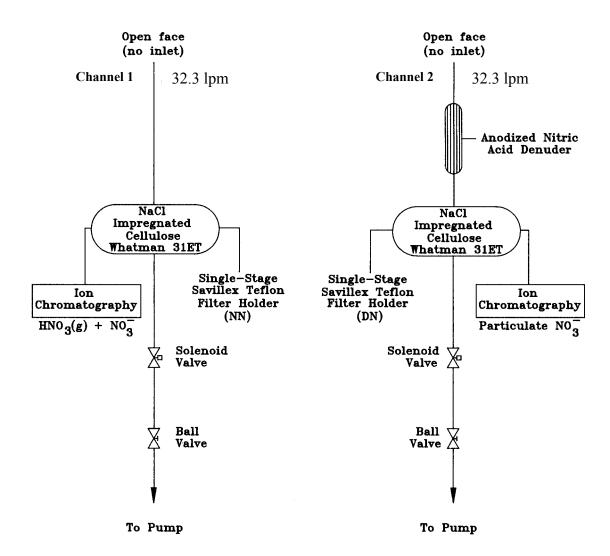


Figure 2-3 Schematic of HNO<sub>3</sub> Sequential Gas Sampling System.

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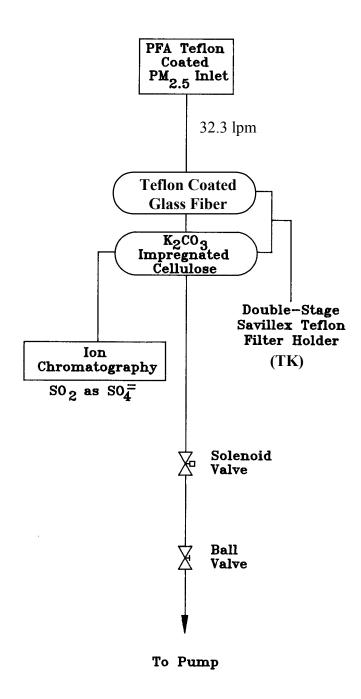


Figure 2-4 Schematic of SO<sub>2</sub> Sequential Gas Sampling System.

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### 2.3.1 Applicability of SOP to HNO<sub>3</sub> Sampling

This SOP is applicable to HNO<sub>3</sub> sampling with the exception that in the case of HNO<sub>3</sub> sampling, filter packs and denuders are not inserted into the bottom of the plenum. Rather, they are placed on a wheel that is mounted on top of the plenum (see Figure 2.b). Other procedures such as the programming of the electronic cabinet and the switching of solenoid valves are accurately prescribed by this SOP.

### 2.4 Sulfur Dioxide Gas

Figure 2.4 shows a schematic of the SGS used for sulfur dioxide sampling. In this case the conical plenum is used in the standard configuration, with only one channel used for sulfur dioxide sampling. A Bendix 240 cyclone coated with PFA Teflon is used as a size-selective inlet at the top of the plenum. The cyclone is used to remove large particles from the particle stream to assist in keeping the plenum clean. A double-staged Savillex Teflon filter holder encloses a Teflon-coated glass fiber filter (TX40 HI20, Pallflex) followed by a potassium carbonate-impregnated cellulose filter (Whatman 31ET). The Teflon filter removes particles from the sample air. The cellulose filter removes sulfur dioxide from the air stream. Depending on expected sulfur dioxide concentrations, two back-to-back cellulose filters, both impregnated with potassium carbonate, may be used to assure efficient removal of sulfur dioxide from the sample air.

### 2.4.1 Applicability of SOP to sulfur dioxide sampling

This SOP is applicable to sulfur dioxide sampling with one exception. During each sample period, sulfur dioxide sampling requires that air be sampled through only one filter pack (as opposed to two filter packs as in the cases of ammonia/ammonium and HNO<sub>3</sub> sampling). Thus, for example, references in the SOP to procedures that apply to "Ports 1 and 7" should be directed to "Port 1" only.

# 3. APPARATUS, INSTRUMENTATION, SUPPLIES, AND FORMS

### 3.1 Apparatus and Instrumentation

### 3.1.1 DRI MEDVOL Particle Sampler.

The sampler is pictured in Figure 2-1. Air enters the inlet, and flows into the conical plenum where filter holders are inserted into the base. The air flows through two of twelve possible filter packs, then through open solenoid valves, differential pressure flow

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controllers, ball valves, and flow rate indicator orifices to one of two GAST 1022/1023 carbon vane pumps. These pumps have sufficient capacity to pull between 50 to 60 lpm through most filter packs and enough additional flow through the makeup air port to produce a total flow rate of 113 lpm.

The differential pressure flow controllers maintain constant pressure, and therefore a constant flow rate, across ball valves for the sampling ports containing filters. This is needed because of the increased resistance caused by filter loading during sampling. As the filter loads up, the pressure drop across this valve decreases which sends a signal to the valve to open further and allow more air to pass. This then equalizes the pressure across the valve.

The makeup flow rate is controlled by a separate valve. Table 3-1 lists each component of the sampler and its function. Literature describing the timer, differential pressure regulator, and pumps are attached to this procedure.

### 3.1.2 Savillex Filter Holders.

The filter holders are open faced and accommodate 47 mm diameter filters. Labels with ID numbers for the filters are attached to the filter holders when the filters are loaded. Plugs for the holders are provided to block the flow when holders are not used.

### 3.1.3 Dwyer 0 to 100 SCFH Rotameter

This rotameter is used to set and verify flow rates through the filter packs. It is fitted with Tygon tubing and a number 9.5 rubber stopper adapter that fits into filter holder receptacle.

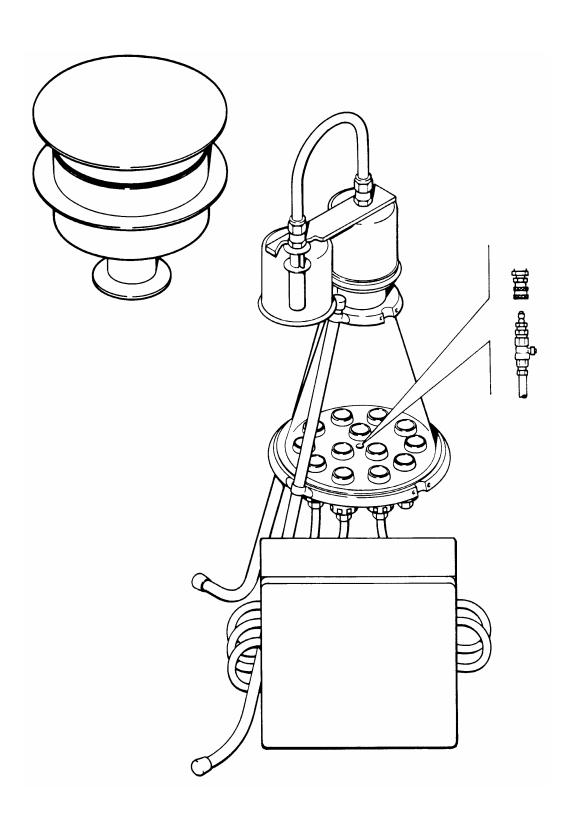
### 3.1.4 Dwyer 0 to 400 SCFH rotameter.

This rotameter is used to measure the makeup air flow rate and to verify the total flow rate into the inlet.

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Figure 3-1. Schematic of DRI MEDVOL Particle Sampler

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### 3.1.5 Total Flow Adapter.

This adapter is placed on the conical plenum in place of the inlet to verify the total flow rate and check for leaks.

### 3.2 Maintenance

### 3.2.1 Change Pump Exhaust Filters

For pumps with the glass jar filters, change the fiber exhaust filter once a quarter. Unscrew the glass jar enclosing the exhaust filter. Grasp the filter (work gloves may be desirable to avoid fiber slivers in the hands), pull downward slightly and unscrew the bottom filter retainer plate. Insert a new filter and finger-tighten the retaining plate. Wipe residue from the glass jar and replace. Change inlet filter once a year. For pumps with cartridge exhaust filters, change the filter once every six months.

### 3.2.2 Clean PM<sub>10</sub> Inlet

Clean inlet every six months or as needed. Remove the Sierra Andersen 254 inlet by loosening the collar and pulling it off. Wipe out the inside of the plenum with a DDW dampened Kimwipe until all soil is removed. Dry it with a clean Kimwipe. Disassemble the inlet by removing the center screw and pulling the top off. Remove the impaction jets by removing the hold-down screws and pulling it out. Earlier versions of this inlet were sealed with silicon sealer. Cut this sealer with a utility knife to remove it. Place flat door insulation around the inside mounting ring when replacing the impactor jets, as this is what has been done with more recent versions of this inlet. With the impaction jets removed, wipe the impaction surface and inside of the inlet with a DDW dampened Kimwipe until all dirt is removed. Scrub out the inside of the impactor jets with a tubular brush (like a test tube brush). Dry all surfaces with clean Kimwipes. If soiling is still observed on the Kimwipes, the inlet is not yet clean.

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### 3.2.3 Clean $PM_{2.5}$ Inlet

Clean inlet monthly. Remove the PM<sub>2.5</sub> inlet assembly from the top of the plenum by loosening the collar and pulling it off. Remove bug screen and cyclone from inlet, taking care not to scratch Teflon coating. Clean inside surfaces of both inlet containers with DDW dampened Kimwipes, and dry with a clean Kimwipe. Clean Teflon tube by rinsing with DDW and dry by pushing a small piece of Kimwipe through with a section of smaller diameter Teflon tubing. Install new cyclone that was cleaned and greased at the laboratory. Make sure the connecting nut holds the cyclone tightly, and that the cyclone has a cap on the bottom. Install bug screen.

### 3.2.4 Clean Plenum

Clean plenum at the beginning and end of the sampling program. Wipe out the inside of the plenum with DDW dampened Kimwipes until all soil has been removed. Dry it with a clean Kimwipe.

### 3.2.5 Change "O"-Rings in Filter Ports

Replace the "O"-rings in the filter ports of the plenum base annually. Remove each "O"-ring with a small pointed object, taking care not to scratch the "O"-ring groove in the sampler, and install a new "O"-ring making sure it is in groove all the way and not twisted. In addition to the annual schedule, replace the "O"-rings if they become hard or cracked or if filter holders become loose in their ports

### 3.3 Supplies

Kimwipes Large size, to clean inlets and plenum.

DDW To clean inlets.

Pump Exhaust Filters GAST #A393 cylindrical exhaust filters or Gelman capsule filter #12116.

"O" Rings To seal and hold filter packs in plenum base.

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# 3.4 Forms

The data sheet for recording field data is shown in Figure 3-2.

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DRI Sequential Filter Sampler Field Data Sheet	11	Titer to							Particle Size: Use I for PM <sub>10</sub> , F for PM <sub>23</sub> , and C for PM <sub>23</sub> to PM <sub>10</sub> Sampling Period: Circle time zone: Pacific (PSI, PDI), Mountain (MSI, MDI), Eastern (ESI, EDI)  Flow rate: Rotameter reading in Standard Cubic Feet per Hour (SCFH)

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Figure 3-2. Example of Data Sheet for Recording Field Data.

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### 4. CALIBRATION STANDARDS

The transfer standards for flow rates are the rotameters specified in section 3.1. They are all calibrated before use against a positive displacement Roots meter, model 1.5M125, serial number 8623119. Elapsed time indicators are checked against a stopwatch.

### 5. SAMPLER OPERATION

### 5.1 Flow Diagram

Figure 5-1 summarizes the routine operating procedure for the DRI MEDVOL. Flow rate checks are made in between intensive sampling periods and a leak check is performed monthly to track sampler performance. Exhaust filters are changed every quarter or six months (depending on type). Every six months or at the beginning and end of a sampling program, the plenum is cleaned; every month, a cleaned and regreased cyclone is installed.

## 5.2 Sampler Installation and Start Up

#### 5.2.1 Installation

Sampler location should follow EPA siting criteria for particulate sampling. The height of the inlet should be 2 to 15 meters above the ground. The distance to horizontal obstacles should be greater than 2 meters and should be at least twice the height that the obstacle protrudes above the sampler. The sampler should be more than 20 meters from trees. There should be unrestricted air flow through 270° around the sampler. No furnace or incinerator flues should be nearby to cause undue influences from minor pollutant sources. A site survey of within 100 meters of the sampling site should be made to determine heights of obstacles along with their distances and directions relative to the sampler. Nearby sources should be documented.

The MEDVOL sampler should be installed on a wooden platform, if possible. The sampler requires one square meter of space for itself along with a work area space of one square meter in front, one square meter to the rear and one square meter to at least one side. The inlet should be separated from the inlets of other samplers by a distance of at least two meters. Make sure inside of plenum and base plate are clean. Place large "O"-ring in groove in base plate and attach plenum to plate. Install small "O"-rings in grooves that are in the holes in the base plate. Attach four legs loosely to plenum with metal conduit straps. Secure the four legs of the

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sampler to the platform with similar metal straps. Even legs on plenum so that base is horizontal and tighten straps.

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Attach control box to two of legs. If external tubing has not been attached, attach 3/8 inch ID soft plastic tubing to solenoid valves on both sides of control box by passing tubing through holes in sides of box. Solenoids 1 - 6 are on the right-hand side while 7 - 12 are on the left-hand side. Secure plastic tubing with hose clamps. Insert three inch-long, 3/8 inch-diameter hard plastic tubes in ends of soft plastic tubing and secure with hose clamps. Score exposed ends of hard tubes with 3/8 inch scoring tool and insert into filter holder nuts. The nuts will stay with the sampler, not the filter holders.

Attach 3/8 inch ID flexible plastic tubing to makeup air flow line through left-hand side of control box. Attach quick disconnect fitting to tubing.

For  $PM_{10}$  sampler, check that Sierra Andersen model 254 size selective inlet is clean. If necessary, clean inlet following instructions in section 2.2.2. Place inlet on top of flange on plenum with gasket in between and attach clamp.

For PM<sub>2.5</sub> sampler, place 1/2 inch Swagelock bulkhead fitting on end of a cleaned and greased cyclone using a nylon ferrule. Install bulkhead fitting through bottom of one of the two inverted Teflon coated containers with cyclone inside. This chamber acts to still the air entering the cyclone. Install a second bulk head connector through the hole in the bottom of the second container with a piece of gasket material around the connector. Attach a 1/2 inch Teflon tube to the two bulk head connectors. Place red end cap on cyclone. Place screen over stilling chamber and attach with small screws. The screen will prevent large bugs from entering the sampler. Place gasket on top of flange on plenum, place second container (the one without cyclone) on gasket and secure with clamp.

Under high wind conditions, it may be necessary to guy the inlets to the platform to prevent their being blown over.

Place the pumps below and as far away from the sampling inlet as possible. Even though the pump exhaust is filtered, it is good practice to have it removed from the sample inlet. If possible, place the pumps underneath the platform. This may require increased length of tubing and electrical cord. Attach 1/2 inch braided plastic tubing to exhaust of sampler and inlet of pump. Plug electrical cords from pump boxes into receptacles on sides of control box. Plug power cord from sampler into 110 VAC power. The sampler requires two separate 15 amp circuits. Turn power switch in control box on.

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### 5.2.2 Set Time on Timer

The time for the sampler is generally Local Standard Time and should remain so throughout the year. The time should be set using a watch known to be accurate to  $\pm 5$  minutes as determined from WWV radio or other means.

To set the time, hold "Set Time" button in while performing the following operations:

- 1. Push day of week button so that LCD bar is above the proper day.
- 2. Push h+ (or h-) button until proper hour is shown (including AM or PM).
- 3. Push m+ (or m-) button until one minute more than current time is shown.
- 4. Release "Set Time" button when minute on watch changes to next value.

The clock will start running when "Set Time" button is released. The current time should be verified on each site visit and re-set if it is not within five minutes of watch time. A built-in rechargeable NiCd battery will maintain the correct time for up to 100 hours in the event of a power outage.

#### 5.2.3 Set Flow Rates.

Follow the calibration procedure described in section 6 to set flow rates for 50 to 60 lpm (as specified by the field operations supervisor) for filters and the appropriate value for a makeup flow rate to total 113 lpm.

### 5.2.4 Check for Leaks.

Remove inlet from plenum and insert the total flow adapter in the top of the plenum. Insert filter holders containing test filters into all open ports in the base of the plenum and connect all sample tubes. Turn on pumps and adjust flowrates to their setpoints. Turn off pumps, attach the 0 to 400 SCFH rotameter to the total flow adapter at inlet of plenum, turn pumps on, and measure total flow rate into plenum. The flow rate through the total flow adapter and the sum of the flow rates through all filter packs plus makeup air port should agree to within 5%. If they do not, then there are excessive leaks in the sampler which must be identified and eliminated. These can occur l) where the filter holders insert into the plenum,

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2) through the filter holders, 3) through the solenoids and manifolds, 4) through any of the tubing and connections up to the valves, or 5) where the rotameter adapter is connected to the plenum. Record the results of the test in the station log and flow calibration sheet.

### 5.2.5 Program Timer

The timer has two channels. Channel 1 turns the pumps on and off. Channel 2 switches the solenoid valves in sequence. Both channels can be turned on and off manually by pushing the respective "OVERRIDE" button. To manually switch solenoid valves using Channel 2, it is necessary to pause several seconds after turning the channel off before turning it back on. Always return Channel 2 to off by pressing "OVERRIDE" again after changing solenoid valves. All operations require two programs: the first to turn a channel on and the second to turn it off. All sets of programs are entered in the following manner:

### Program 1:

- 1. Push timer button corresponding to day that sample will start.
- 2. Push +h (or -h) to display starting hour. Make sure AM or PM is correct.
- 3. Push +m (or -m) to display starting minute.
- 4. Push I/O button once to display a bar below I (for ON) for desired channel.
- 5. Push "WRITE" to save this program.

### Program 2:

- 6. Push timer button corresponding to day that sample will end.
- 7. Push +h (or -h) to display ending hour. Make sure AM or PM is correct.
- 8. Push +m (or -m) to display ending minute.
- 9. Push I/O button twice to display a bar below O (for OFF) for desired channel.
- 10. Push "WRITE" to save this program.

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### **5.2.5.1** To review programs

First push "SET TIME" to return current time display. Then push "READ" to display first stored program. Each subsequent push of "READ" displays another program until all have been displayed. All programs should be reviewed each site visit and particularly after any programs are added, modified, or deleted.

### 5.2.5.2 To change a program

First display program to change by pushing "READ". Then enter desired change. Note that to change day, the button for the old day has to be pushed to remove the bar for that day. After the new day and time are displayed, push "WRITE" to save the new program. Both the original program and the changed program are now in memory. Delete the original program (see below) if it is no longer needed. To produce a series of similar programs, make changes to the original program as described above, without deleting original program, and repeat until all programs have been changed. All changes should be reviewed.

### 5.2.5.3 To delete a program

First display the program to delete by pushing "READ". Then press the "CANCEL" button to delete the program. Full programming instructions are appended to these procedures.

### 5.2.5.4 Programming the Start and Stop times for Channel One:

Note that because the Treasure Valley Secondary Aerosol study involves intensive sampling for intervals of unknown length (anywhere between 1 and 10 days), it is not always possible to determine in advance when the stop time should be for a sampling interval. Thus, the stop time should be programmed for exactly one week after the start. Once the actual stop time (specifically, the day of the week) is known, the Channel 1 program may be changed accordingly.

Push the "READ" and "CANCEL" buttons several times in succession until a blank display appears. This clears all previous programming steps. Push "Set Time" and the timer is prepared for programming. To program the SGS to start sampling at midnight, push the button corresponding to the days of the week until the bar on the D display appears over the correct day of the week. Note that if you are programming to start at midnight, the start day for the sample is actually tomorrow, i.e. if you are entering the program on Tuesday for a

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midnight start, then the start day is a Wednesday. To program the start time, push the "h+" button until 12 am is displayed in the hours column. Push the "m+" button until 01 is displayed in the minute column. Press the Channel 1 "I/O" button until the Channel 1 LCD marker is on "I". Press the "WRITE" button to record this program step. To program the stop time, press the appropriate buttons as above to get a stop time of 11:59 PM ON THE PREVIOUS DAY OF THE WEEK as the start time. So, if the timer is set to start at 12:01 AM on Wednesday, the stop time should be set for 11:59 PM on Tuesday. Press the Channel 1 "I/O" button until the display reads "O". Press the "WRITE" button to record this

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When the actual stop date for intensive sampling becomes known, program the channel 1 stop time so that channel 1 is set to "O" at 11:59 PM on the last day of sampling.

Note that there are three SGS and one SFS samplers running at each of the two sites selected for the Treasure Valley study. Since these samplers will all be operating concurrently, it is important to space out the times that the pumps turn on in order to avoid tripping circuit breakers. Thus, the SFS samp;ler should be programmed to turn on at 12:00 am, the Ammonia/ammonium SGS at 12:01 am, the nitric acid/total nitrate SGS at 12:02 am, and the sulfur dioxide SGS at 12:03 am.

### **5.2.5.5** Program the Filter Switching Times for Channel 2:

When sampling through one set of filters is complete, a one-minute electrical signal is sent to the appropriate solenoid valves by Channel 2 on the timer. This signal advances the solenoid valve to the next set of ports. Thus, if sampling through ports 1 and 7 starts at midnight and ends at 5:00 AM, a signal must be programmed for 5:00 AM so that sampling stops through ports 1 and 7 and starts through ports 2 and 8. To program an electrical signal for 5:00 AM, press the orange button which corresponds to the desired sampling day until a bar appears over each day of the week. Press "h+" and "m+" until 4:59 AM is displayed. Press the Channel 2 "I/O" button until an "I" appears for channel 2. press "Write" to enter this program step into the timer. Using the orange button, "h+", and "m+" repeat this procedure so that the time is 5:00 AM. This time, press the channel 2 "I/O" button so that an "O" appears on the display for channel 2. Press "WRITE" to enter this program step. This last sequence of steps will send a one-minute electrical pulse to the solenoid valve starting at 4:59 AM, causing the solenoid valve to advance to the next set of sampling ports. Repeat this step for all sample change times. For the Treasure Valley study, there should be a one-minute signal programmed for 5:00 AM, 10:00 AM, 2:00 PM, and 7:00PM, and 12:00 AM.

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# 5.2.5.6 The filter switching program for the Treasure Valley Study

Press "read" to sequence through each step in the program in order. When a change is desired in a step, the appropriate buttons can be pressed to make that change and it will be recorded by pressing "WRITE". Pressing "READ" will start at step number one when it is pressed following a "WRITE". For example, the program entered should read as follows if sampling is to start on Tuesday at midnight:

R SFS TIM	ER PROGRAM 3Y	CH1	CH2
i.	11:59 pm Mo	1	
ii.	04:58am TuWeThFrSaSu		0
iii.	04:59am TuWeThFrSaSu		1
iv.	05:00am TuWeThFrSaSu		0
V.	09:58am TuWeThFrSaSu		0
vi.	09:59am TuWeThFrSaSu		1
vii.	10:00am TuWeThFrSaSu		0
viii.	01:58pm TuWeThFrSaSu		0
ix.	01:59pm TuWeThFrSaSu		1
Χ.	02:00pm TuWeThFrSaSu		0
xi.	06:58pm TuWeThFrSaSu		0
xii.	06:59pm TuWeThFrSaSu		1
xiii.	07:00pm TuWeThFrSaSu		0
xiv.	11:58pm TuWeThFrSaSu		0
XV.	11:59pm TuWeThFrSaSu		1
xvi.	12:00am MoTuWeThFrSaSu		0

Push READ n times to read step n

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Push WRITE after you modify a step and are ready to enter it into the program

Push CANCEL to turn a step into a blank

LEAVE CHANNEL 2 OFF!!!

### **5.3** Sampler Operations

The normal operation of the MEDVOL sampler consists of installing and removing filters, checking flow rate indicators before and after sampling, recording the elapsed time readings, and performing periodic flow rate checks. Samples are delivered to the site or site operator with field data sheets that specify sample IDs and sampling date and time.

Remove filters as soon after sampling as possible to reduce loses of volatile species such as ammonium nitrate and some organic carbon compounds. After removal, refrigerate the samples until they are analyzed to reduce loses. In some programs, it is desirable to install filters the day before sampling and to remove them the day after sampling. The installation and removal procedures are detailed in the following sections.

Manual control of the vacuum pumps and solenoids during sample loading and unloading is accomplished by pressing the "OVERRIDE" buttons on the timer for Channel 1 and Channel 2, respectively. When selecting solenoids, always observe the following instructions: 1) to manually switch solenoid valves using Channel 2, pause several seconds after turning the channel off before turning it back on, and 2) always return Channel 2 to off by pressing "OVERRIDE" again after switching the solenoid valves.

### **5.3.1** Installation of Filters

Sample changes for the Treasure Valley study will occur during the 10:00 AM to 2:00 PM sampling interval. Because the Treasure Valley study requires the collection of five samples in a twenty-four hour period, and because intensive sampling may continue over a period of several days, it is necessary to use all ports for sampling. This means that it will not be possible to collect dynamic field blanks in the middle of an intensive sampling period. However, it is possible to obtain one set of field blank filter packs on the first day of intensive sampling and another set on the last day of intensive sampling. The following tables outline the filter change schedule:

On the day before sampling starts, filter packs are loaded as follows.

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Day 0 (the day before an intensive sampling period is going to be started):

Port(s)	Sample in ports will run:	Filter pack installed on:
1 and 7	Day 1: 12:00 am to 5:00 am	Day 0
2 and 8	Day 1: 5:00 am to 10:00 am	Day 0
3 and 9	Day 1: 10:00 am to 2:00 pm	Day 0
4 and 10	Day 1: 2:00 pm to 7:00 pm	Day 0
5 and 11	Day 1: 7:00 pm to 12:00 am	Day 0
6 and 12	Field Blanks	Field Blanks

The first sample change occurs between 10:00 am and 2:00 pm on Day 1. All ports that have completed sampling and the ports with the field blanks receive unexposed filters. After the first sample change, the SFS should have the following configuration.

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Day 1

Port(s)	Sample in ports will run:	Filter pack installed on:
1 and 7	Day 2: 5:00 am to 10:00 am	Day 1 (10:00am 2:00pm)
2 and 8	Day 2: 10:00 am to 2:00 pm	Day 1 (10:00am 2:00pm)
3 and 9	Day 1: 10:00 am to 2:00 pm	Day 0
4 and 10	Day 1: 2:00 pm to 7:00 pm	Day 0
5 and 11	Day 1: 7:00 pm to 12:00 am	Day 0
6 and 12	Day 2: 12:00 am to 5:00 am	Day 1 (10:00am 2:00pm)

On the sample change on Day 2, all ports that have completed sampling receive unexposed filters. After the sample change on Day 2, the SFS will have the following configuration:

Day 2

Port(s)	Sample in ports will run:	Filter pack installed on:
1 and 7	Day 3: 10:00 am to 2:00 pm	Day 2 (10:00am 2:00pm)
2 and 8	Day 2: 10:00 am to 2:00 pm	Day 1 (10:00am 2:00pm)
3 and 9	Day 2: 2:00 pm to 7:00 pm	Day 2 (10:00am 2:00pm)
4 and 10	Day 2: 7:00 pm to 12:00 am	Day 2 (10:00am 2:00pm)
5 and 11	Day 3: 12:00 am to 5:00 am	Day 2 (10:00am 2:00pm)
6 and 12	Day 3: 5:00 am to 10:00 am	Day 2 (10:00am 2:00pm)

On the final day of sampling, say Day 4, a field blank is loaded into a port that will not sample again till the end of the intensive sampling period. In the case of a Day 4 stop time, the configuration of the SFS will be as follows after the Day 4 (final) sample change:

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Day 4

Port(s)	Sample in ports will run:	Filter pack installed on:
1 and 7	Day 4: 2:00 pm to 7:00 pm	Day 4 (10:00am 2:00pm)
2 and 8	Day 4: 7:00 pm to 12:00 am	Day 4 (10:00am 2:00pm)
3 and 9	Day 4: Dynamic field blank	Day 4 (10:00am 2:00pm)
4 and 10	Plugged ports	Day 4 (10:00am 2:00pm)
5 and 11	Plugged ports	Day 4 (10:00am 2:00pm)
6 and 12	Day 4: 10:00 am to 2:00 pm	Day 3 (10:00am 2:00pm)

It is important to construct a table like the ones shown above for evey day of sampling to minimize the chances of an error occurring during sample changes.

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To perform a sample change, open the plastic bag of new filters, remove their bottom cap, and place them in the sampling port for each ID as required by the schedule. Remove the top cap after the filter holders are in place. If a field blank cassette is included, it should be placed in a port that is not expected to be sampled through. At the beginning of an intensive sampling period, this will be ports 1 and 7. To plug a filter holder into a quick-connect fitting, insert the barb into the fitting. Next, grasp the ring around the fitting and pull it down from the filter holder. Push the barb into the fitting. Release the ring on the fitting and push the filter holder toward the barb until a snap is heard as the ring moves toward the filter holder.

### 5.3.1.1 Check Timer

Open control panel door and compare time on timer to time on accurate watch. Reset time if error is more than five minutes, and note change on field data sheet.

### 5.3.1.2 Inspect Unexposed Filters.

As each filter holder is installed, inspect each one for obvious foreign material on the filter and inside the holder receptacle, and for tears or misalignment of the filter. Replace those which do not pass inspection with one of the holders designated as a spare, if available, or with a field blank. Change sample ID on the field data sheet accordingly.

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# 5.3.1.3 Install Unexposed Filters.

Remove the filter packs from their containers (Ziploc bags). Remove the small bottom cap and then the large top cap by gently prying the side of the cap up until it is off. This cap removal method prevents possible filter damage. Return top and bottom caps to the Ziploc bag and seal it to keep them clean. Check that filter holder receptacle is snug but do not over-tighten. Attach the appropriate tube for the port that the filter pack is to be placed in to the bottom of filter holder and install holder in plenum by pushing the receptacle into appropriate port in bottom of the plenum until the knurled portion of the receptacle comes into contact with the plenum base.

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Similarly install remaining holders in plenum. Install field blanks if they are listed on the field data sheet. After all samples are installed, double check that sample IDs, port numbers, and air flow tube numbers match the field data sheet. Check that unused ports have filter holders with plugs installed.

<u>CAUTION.</u> It is essential that you double check IDs as indicated above EVERY time you load samples. Loading errors cannot be identified or corrected after sampling is completed.

### 5.3.1.4 Set Pre-Exposure Flow Rates

Open the sampler control box door all the way. Turn pumps on if not already on. A light on the stepper switch box indicates which ports are activated. If necessary, activate stepper switch to open ports 1, and 7. Set flow rates on ports 1 and 7 (port 1 only for  $SO_2$  sampler). Record flowrates on field data sheets. This is also a good time to ensure that the correct filterpack is attached to the correct port #.

- a.  $\mathrm{NH_3/NH_4}^+$  sampler: 70 SCFH through filterpacks and 105 SCFH through makeup port
- b.  $SO_2$  sampler: 70 SCFH through filterpack and 175 SCFH through makeup port
- c. HNO<sub>3</sub>/NO<sub>3</sub> sampler: 70 SCFH through the filterpacks. NO NEED FOR MAKE-UP FLOW
- d. Fine particle sampler (SFS): 122.4 SCFH through filterpacks and 0 SCFH through make-up flow port

Adjust flows for the makeup air port first (if applicable), and then ports 1 and 7 by adjusting the ball valves until the flowrate measured by the rotameter is the design flowrate. If one channel requires a large adjustment, other channels may require readjustment. Record all

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flowrate readings on the field data sheet in the "Initial Flow Rate" column to the nearest SCFH.

Switch to ports 2 and 8. Do not adjust flows; they will be slightly different than for the first set of ports. Record all flowrates in the "Initial Flow Rate" column to the nearest SCFH. Continue this procedure for all sets of ports through which samples will be acquired.

Remember to switch to the correct ports for sampling. If you are preparing for the beginning of an intensive sampling period, then the correct ports are 1 and 7. If you are changing samples in the middle of an intensive study, then make sure you leave the same ports on as when you showed up at the site.. <u>DOUBLE CHECK that the proper ports are sampling by observing that the timer for those ports is rotating. If you are preparing to start an intensive sampling period, switch the pump off; if you are changing samples, leave the pump on so that the samples that are currently running complete their sampling schedule. <u>DOUBLE CHECK that Channel 2</u> is off.</u>

## 5.3.1.5 Record Initial Elapsed Time

Record the elapsed time indicator readings on the field data sheet in the "Elapsed Time Start" box.

### **5.3.1.6** Verify Programs

Verify that all programs are correct by pressing "READ" button successively until all programs are reviewed. If any changes are made, verify the changes. Record timer day and date of sample loading on a blank line of the field data sheet.

#### **5.3.2** Removal of Filters

# 5.3.2.1 Inspect MEDVOL Sampler and Record Final Elapsed Time

Open control panel door and compare time on timer to time on accurate watch. If more than five minutes off, note discrepancies on field data sheet. Record timer day and date of sample unloading on field data sheet. Record elapsed time indicator values in the "Elapsed Time End" space. Calculate the sample elapsed time and record on field data sheet. It

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should be within a few minutes of the scheduled time. Note any discrepancies on the data sheet.

### **5.3.2.2** Record Post-Exposure Flowrates

Open the sampler control panel door all the way. Turn pumps on (they will already be on if this is occurring in the middle of an intensive sampling interval). Check that open ports are the expected ones for the last sample or for the sample that is currently running. Record flowrates for all ports that have sampled on field data sheet in "Final Flow Rate" column. Switch ports to open 1 and 7. Turn pump off.

### **5.3.2.3** Remove Exposed Filters

Ensure that pump is turned off before removing filter holders. Remember that if this is a sample change (occurring between 10:00 am and 2:00 pm), two of the filter holders will still be sampling. These SHOULD NOT BE REMOVED. Remove the exposed filter holders by pulling them from the plenum one at a time. Verify that filter ID on filter pack, port number, and flow tube number match the information on the field data sheet; note any discrepancies. Remove top and bottom caps from the Ziplock bag, check that they are clean, place them on filter holder, and reseal filter pack in Ziplock bag. Place the top cap on before the bottom cap to prevent pressure buildup in the filter holder caused by compressing air under the cap.. When all filters are unloaded, place them in refrigerated storage.

### 5.3.3 Return Samples

Return collected samples monthly to DRI. Place all sampled filters including field blanks in a cooler with Blue Ice. Enclose field data sheets and flow check data in Ziplock bag. Ship as instructed by field operations supervisor.

#### **5.3.4** Flow Rate Performance Test and Leak Check

Conduct a flow rate performance test and leak check once a month. Load a set of test filter packs in ports 1 and 7 and plugged filter packs in the remaining ports. Turn on pumps and adjust flow valves so that the flowrates through Ports 1, 7, and the "make-up air" port are set to their design values. Remove inlet and place total flow adapter in top of plenum. Connect 0 - 400 SCFH rotameter to the total flow adapter, turn on pumps with ports 1, 7, and the make up air port running and measure total flow rate. Make sure that rotameters are

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level when taking readings. Record all Magnehelic and rotameter readings as they are taken.

### 5.4 Shutdown

At the end of the sampling program, conduct a leak check and flow performance test. Record the condition of the sampler in the station logbook.

# 6. QUALITY CONTROL

### 6.1 Calibration Checks

Flow rate performance checks as described in Section 5.2.3 are made monthly.

### 6.2 Leak Checks

A leak check as described in Section 5.2.4 is performed monthly.

### 6.3 QUALITY AUDITING

Audits of flow rates are performed by an independent auditor with independent standards on a regular schedule, usually twice a year.

### 7. REFERENCES

NOAA, 1976. "U. S. Standard Atmosphere, 1976" by the National Oceanic and Atmospheric Administration, Washington, DC, Oct. 1976.